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14. ABSTRACT

This report summarizes the progress and achievements resulting from the Army Research Office grant number WF911NF-08-1-0350 on the subject of High Resolution Orientation Imaging Microscopy. The central technical outcome of the project is a new methodology for extracting high-resolution structure data from crystalline materials that uncovers a wealth of new microstructure information including absolute strain, geometrically necessary dislocations, and improved orientation measurements. The framework has been patented, and a commercialization

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High Resolution Orientation Imaging Microscopy: Final Report

ABSTRACT

This report summarizes the progress and achievements resulting from the Army Research Office grant number WF911NF-08-1-0350 on the subject of High Resolution Orientation Imaging Microscopy. The central technical outcome of the project is a new methodology for extracting high-resolution structure data from crystalline materials that uncovers a wealth of new microstructure information including absolute strain, geometrically necessary dislocations, and improved orientation measurements. The framework has been patented, and a commercialization agreement is in place to disseminate the new capabilities to the scientific community. New insights into deformation processes in magnesium, nickel, tantalum, iron, copper and various other metals have already arisen. The results have been widely published via journals, books and conferences.
Four graduate students have been funded by this grant; two have earned PhDs, and two are on track to complete PhDs. Three students have been awarded NSF or NDSEG fellowships as a direct result of the research opportunities afforded by this grant to undergraduate students. Various other undergraduate students have been funded and have moved on to graduate school.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

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1. D T Fullwood, Tim Ruggles, Travis Rampton, Thoughts on rationalizing a continuum dislocation model with increasingly high resolution characterization and modeling tools, Plasticity, Nassau, 2013 (keynote)
2. B.L. Adams, D.T. Fullwood, High Resolution EBSD-Based Micromechanical Stereoinference, APM2012 Summer School-Conference, St Petersburgh, 2012
3. D T Fullwood, Tim Ruggles, Travis Rampton, Michael Miles, Raj Mishra, Insights from Lattice Gradient Measurement with High Resolution EBSD, MS&T, Pittsburgh, 2012 (invited)
4. D T Fullwood, Travis Rampton, Tim Ruggles, High Resolution EBSD as an Input to Structure Property Relations, MS&T, Pittsburgh, 2012 (invited)
5. D.T. Fullwood, S. Subedi, Measuring Lattice Strain with High Resolution EBSD, EBSD, Pittsburgh, 2012 (keynote)
6. David Fullwood, Tim Ruggles, Ali Khosravani, Mike Miles, Raj Mishra, Dislocation Studies: Extracting Knowledge From EBSD, Int. Conf. Plasticity, Puerto Rico 2012 (keynote)
7. Brent L. Adams, David T. Fullwood, Thomas Harding, Jay Basinger, High Resolution EBSD-Based Dislocation Microscopy, 2011, Los Alamos Center for Integrated Nanotechnologies (invited)
8. David T. Fullwood, Brent L. Adams, Jay Basinger, Travis Rampton, Ali Khosravani, Tools for Investigating Localization and Damage in Polycrystalline Materials, 2011, Los Alamos Center for Integrated Nanotechnologies (invited)
9. Brent L. Adams, David T. Fullwood, Thomas Harding, Jay Basinger, Nachiket Patil, High Resolution EBSD-Based Microscopy for Damage Detection, USNCCM 2011, Minneapolis
10. Brent L. Adams, Thomas Hardin and David T. Fullwood, Recovering the full dislocation tensor from high-resolution EBSD microscopy, ICHMM, 2011
11. Calvin Gardner, Brent Adams, David Fullwood, Extending the Effective Range of Wilkinson’s Method Via a Geometry-Based Pattern Center Correction Algorithm, TMS, San Diego, 2011
12. Brent Adams, Samikshya Subedi, Sadegh Ahmadi, David Fullwood, Robert Wagoner High-Resolution EBSD Characterization and Analysis of Defect Structure of In-Situ Deformations of Steel, TMS, San Diego, 2011
13. Jay Basinger, David Fullwood, Brent Adams, EBSD Detail Extraction for Greater Spatial and Angular Resolution in Material Characterization, TMS, San Diego, 2011
14. Ali Khosravani, Brent L. Adams, David T. Fullwood, Mike Miles, Stuart Rogers, Jonathan Scott, Utilizing HR-OIM and In-Situ Tensile Tests for Studying Crack Initiation in AZ31 Magnesium Alloys, TMS, San Diego, 2011
15. B.L. Adams, D.T. Fullwood, J. Basinger, T. Hardin, HR-EBSD Microscopy for Localization Studies: Opportunities and Challenges, Int. Conf. Plasticity, Puerto Vallarta, Mexico, 2011
16. Seegmiller, Daniel; Fullwood, David T.; Adams, Brent L., Microstructural Defect Analysis Using Semi-Infinite Green’s Functions, ICCES 2010, Las Vegas.
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18. Adams, B.L., and D.T. Fullwood, An Experimental Approach to the Study of Localization in Polycrystals, Plasticity 2010, St Kitts (keynote).
19. Adams, B.L., High Resolution EBSD Methods for the Study of Grain Boundary Elastic/Plastic Properties, EBSD, Derby, 2010
20. Johnson, O. K., Kaschner, G. C., Mason, T. A., Fullwood, D. T., Hyatt, T., Adams, B. L. and Hansen, G., Extreme piezoresistivity of silicone/nickel nanocomposite for high resolution large strain measurement, TMS 2010, Seattle.
21. Johnson, O. K., Mara, N., Kaschner, G. C., Mason, T. A., Fullwood, D. T., Adams, B. L. and Hansen, G., Multi-scale Model for the Extreme Piezoresistivity in Silicone/Nickel “Nanostrand”/Nickel Coated Carbon Fiber Nanocomposite, TMS 2010, Seattle.
22. Brent Adams, David Fullwood, Microstructure Design for Extreme Value Properties, MS&T. 2009: Pittsburgh. (Invited)
23. Gardner, C., Brent Adams, David Fullwood, Josh Kacher, Scott Lemon, Dan Seegmiller Jay Basinger, Correlation of Grain Size with Dislocation Density in Polycrystals: , in MS&T. 2009: Pittsburgh.
24. Sadegh Ahmadi, Brent Adams, David Fullwood, A New Double Continuity Relation for Predicting the Evolution of Pair Correlation Statistics with Viscoplastic Deformations, MS&T. 2009: Pittsburgh.
25. John Basinger, David Fullwood, Brent Adams, Josh Kacher, Improving Spatial and Grain Boundary Inclination Resolution through EBSD Pattern Separation and Deconvolution, MS&T. 2009: Pittsburgh.
26. Josh Kacher, Brent Adams, David Fullwood, Detection of Tetragonalities and Pseudo-Symmetries by High Resolution EBSD Methods, MS&T. 2009: Pittsburgh.
27. Oliver Johnson, Calvin Gardner, David Fullwood, Brent Adams, George Hansen, Surya Kalidindi, Textures of Dispersion of Nickel Nanostrand Composites, and Modeling of Piezoresistive Behavior: , MS&T. 2009: Pittsburgh.
28. Seegmiller, D.B., D.T. Fullwood, and B.L. Adams, Semi-Infinite Green’s Functions Localization and Defect Detection Applications, MS&T. 2009: Pittsburgh.
29. Gardner C.J., Johnson O.K., Hansen G., Adams B.L., Fullwood D.T., Colossal Piezoresistive Effect in Nickel Nanostrand - Polymer Composites, TMS, San Francisco, 2009
30. Brent L. Adams, Brad Fromm, Ribeka Takahashi, Spectral Four-Parameter Microstructure Design: Observable Local State Spaces, Plasticity 2009, US Virgin Islands

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Received	Paper
05/01/2012	28.00 David Fullwood, Brent Adams, Travis Rampton , Ali Khosravani , Michael Miles, Jon Scott, Raj Mishra. Intelligent microscopy for EBSD applications, ICOTOM 2011. 2011/09/20 02:00:00, . : ,
05/01/2012	34.00 David T. Fullwood, Sadegh Ahmadi, Brent L. Adams. Spectral formulation of statistical theory of viscoplasticity, Plasticity 2009. 2009/01/05 02:00:00, . : ,
05/01/2012	33.00 Oliver K. Johnson, C.J. Gardner, David T. Fullwood, Brent L. Adams, George Hansen. Deciphering the Structure of Nano-Nickel Composites, SAMPE 2009. 2009/05/21 02:00:00, . : ,
05/01/2012	31.00 Oliver K. Johnson, David T. Fullwood. A percolation/quantum tunneling model for the unique behavior of multifunctional Silicone/Nickel Nanostrand nanocomposites, SAMPE Technical Conference 2010. 2010/09/21 02:00:00, . : ,
05/01/2012	30.00 David Fullwood, Brent Adams, Travis Rampton, Ali Khosravani. Intelligent Microscopy for the Study of Fracture and Fatigue, TMS 2011. 2011/02/20 02:00:00, . : ,
05/01/2012	29.00 Oliver K. Johnson, Daniel Seegmiller, David T. Fullwood, Andrew Dattelbaum, Nathan A., Mara, George Kaschner, Thomas Mason. Characterization of electrical properties of polymers for conductive nano-composites, SAMPE 2011. 2011/05/22 02:00:00, . : ,
10/17/2011	7.00 B.L. ADAMS, D.T. FULLWOOD, J. BASINGER, T. HARDIN. High Resolution EBSD-Based Dislocation Microscopy, ICOTOM 2011. 2011/12/17 02:00:00, . : ,
10/17/2011	8.00 Thomas Hardin, Brent L. Adams, David T. Fullwood, Robert H. Wagoner. Estimation of the Full Nye Tensorby EBSD-Based Dislocation Microscopy, ICOTOM 2011. 2011/12/17 02:00:00, . : ,

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Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received		Paper
05/01/2012	23.00	J.A. Basinger, C.J. Gardner, D.T. Fullwood, B.L. Adams. Pattern Center Sensitivity for Simulated Reference Patternsin High Resolution Electron Backscatter Diffraction, Microscopy and Microanalysis (01 2012)
05/01/2012	27.00	Sadegh Ahmadi, Brent L Adams, David T Fullwood. Microstructure Evolution in Polycrystalline Materials: Formulation and Application of a NewEulerian Continuity Model, International Journal of Plasticity (11 2011)
05/01/2012	26.00	T.J. Ruggles, D.T. Fullwood. Estimations of Bulk Geometrically Necessary Dislocation Density Using High Resolution EBSD, Ultramicroscopy (03 2012)
05/01/2012	25.00	T.J. Ruggles, T.M. Rampton, S.A. Rose, D.T. Fullwood. , Reducing the Microstructure Design Space of 2nd Order Homogenization Techniques Using Discrete Fourier Transforms, Mechanics of Materials (04 2012)
05/01/2012	24.00	J.A. Basinger, C. Sorensen, D.T. Fullwood, M.M. Nowell. Full Grain Boundary Character Recovery from 2D EBSD Data, METALLURGICAL AND MATERIALS TRANSACTIONS A (05 2012)
10/17/2011	5.00	J.A. Basinger, C.J. Gardner, D.T. Fullwood, B.L. Adams. Pattern Center Sensitivity for Simulated Reference Patterns in High Resolution Electron Basckscatter Diffraction, Microscopy and Microanalysis (10 2011)
10/17/2011	6.00	Timothy Ruggles, David Fullwood. Estimation of bulk dislocation density based on known distortion gradients recovered from EBSD, Scripta Materialia (10 2011)
TOTAL:		**7**

Number of Manuscripts:

Books

Received		Paper
05/01/2012	11.00	David T. Fullwood, Surya R. Kalidindi, Brent L. Adams. Electron Backscatter Diffraction in Materials Science: Chapter 12, Boston, MA: Springer US, (12 2009)
05/01/2012	10.00	Surya R. Kalidindi, David T. Fullwood, Brent L. Adams. Electron Backscatter Diffraction in Materials Science: Chapter 11, Boston, MA: Springer US, (12 2009)
05/01/2012	17.00	Brent Adams, Surya Kalidindi, David Fullwood. Microstructure Sensitive Design for Performance Optimization, Waltham, Massachusetts: Butterworth-Heinemann, (07 2012)
05/01/2012	18.00	David Fullwood, Brent Adams, Jay Basinger, Timothy Ruggles, Ali Khosravani, Caroline Sorensen, Joshua Kacher. Microstructure Detail Extraction via EBSD: An Overview, Chapter in: Strains and dislocation gradients from diffraction, London: Imperial College Press, (12 2012)

TOTAL: **4**

Patents Submitted

Systems and methods for determining crystallographic characteristics of a material

Patents Awarded

Awards

Brent Adams, Bunge Award, ICOTOM 16, Mumbai, India, December 2011.
Brent Adams, Governor’s Medal of Science and Technology, State of Utah, 2008.
Brent Adams, symposium in honor of Brent Adams' 60th Birthday, MS&T 2009.

Graduate Students

NAME	PERCENT_SUPPORTED	Discipline
Sadegh Ahmadi	0.25	
Joshua Kacher	1.00	
John Basinger	1.00	
Timothy Ruggles	0.10	
FTE Equivalent:	**2.35**	
Total Number:	**4**	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

NAME	PERCENT_SUPPORTED	National Academy Member
David Fullwood	0.13	
Brent Adams	0.13	
FTE Equivalent:	**0.26**	
Total Number:	**2**	

Names of Under Graduate students supported

NAME	PERCENT_SUPPORTED	Discipline
Dan Koch	0.20	Mechanical Engineering
Thomas Hardin	0.20	Mechanical Engineering
Oliver Johnson	0.25	Mechanical Engineering
Calvin Gardner	0.25	Mechanical Engineering
Samikshya Subedi	0.30	Mechanical Engineering
Suzu Prasad	0.50	Mechanical Engineering
Matthew Converse	0.10	Mechanical Engineering
Matthew Hutchinson	0.10	Mechanical Engineering
Dan Seegmiller	0.30	
FTE Equivalent:	**2.20**	
Total Number:	**9**	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 6.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:...... 6.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:...... 6.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):...... 6.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 4.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:...... 4.00

Names of Personnel receiving masters degrees

NAME	
Joshua Kacher	
Total Number:	**1**

Names of personnel receiving PHDs

NAME	
John Basinger	
Sadegh Ahmadi	
Total Number:	**2**

Names of other research staff

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

See Attachment

Technology Transfer

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1 Statement of the Problem Studied

Orientation Imaging Microscopy (OIM) has been an essential tool in the analysis, design and development of crystalline materials since its introduction in the early 90's by one of the PIs and collaborators. Its use has contributed to the development of new steels, aluminum alloys, high T_C superconductors, electronic materials, lead-free solders, optical prisms, etc. and has found applications in many other material systems. However, the basic algorithms used in the OIM framework lead to limitations in resolution that restrict application in the rapidly expanding field of nano-materials.

This project successfully sought to develop a suitable high resolution OIM (HROIM) and demonstrate both its viability and its effectiveness using a variety of test-beds. HROIM is based on rapid analysis of Electron Back-Scatter Diffraction (EBSD) patterns. The desired additional functionality over OIM included: high angular resolution at large and small misorientations; nano-scale feature resolution; resolution of elastic lattice distortion; and phase separation for lattice transformations of small degree.

The envisaged technical approach involved three main steps: 1) accurate determination of orientation and strain relating to a single EBSD pattern; 2) resolution of contributing patterns from an image comprising multiple crystal structures within the instrument probe volume; 3) deconvolution of the resultant multi-state information to provide accurate representation of spatial structure.

Step 1) built upon work by Angus Wilkinson's group at Oxford University, using cross-correlation of EBSD patterns to recover lattice orientation and disorientation with high angular resolution (0.0050). This refined angular precision also enables recovery of the full local elastic strain field tensor, to a precision of 10^{-4}. Simulated patterns introduced by the BYU team provided several significant advantages in these measurements once the angular resolution of this method was improved. The resolution of coincident patterns (step 2) proceeded in parallel with the previous step, using convolution methods to match the different patterns, and determine their contribution to the image. Step 3) then proceeded, based upon deconvolution methods, to resolve the spatial information to higher accuracy.

Finally, rapid algorithms, based upon Fast Fourier transforms were developed to approach the full automation in area scanning that has made OIM a powerful tool. These algorithms have been developed in conjunction with EDAX, under the scope of a commercialization agreement, in order to make the HROIM tools available to the general scientific community.

The capabilities and functionality of HROIM has been evaluated against a test bed of various metal alloys. These include magnesium, nickel, tantalum, copper, and others. More details will be given below.

2 Summary of the Most Important Results

Key points of scientific progress include the following:

1. A novel pattern center determination method (central to achieving high accuracy) has been extensively validated, coded, and applied to strain measurements with dramatic reduction in error

2. The simulated pattern method was implemented to enable the extraction of ABSOLUTE orientation and strain measurements, not previously available
3. Recovery of lattice tetragonality and pseudo-symmetry resolution has been demonstrated
4. Higher accuracy dislocation density information has been extracted via HROIM, and correlations with twin nucleation have been observed
5. Determination of the full Nye tensor from 2D information was demonstrated, and an improved algorithm for determining relative activity of slip systems was tested on simulated fields
6. Improved spatial resolution has resulted from segregation of merged patterns and 3D grain boundary information has been recovered from 2D data
7. Commercially-ready HROIM code has been shown to improve key OIM error measures by more than an order of magnitude
8. Undergraduate research funded by this project has also developed piezo-resistive nano-composites for strain measurement, and studied them using various novel techniques

These points will be reviewed in more detail below.

2.1 Accurate Determination of EBSD Pattern Center

The pattern center measurement determines the displacement between the point on the sample impacted by the electron beam, and the perpendicular point on the phosphor screen. In ordinary EBSD-based methods, like Orientation Imaging Microscopy (OIM), this measurement is determined to within tens of microns using a periodic calibration. However, this accuracy is inadequate for HROIM. A more accurate measurement is required for absolute orientation and strain measurements.

The BYU team has developed (and applied for a patent for) a new approach to pattern center calibration that provides accurate measurements using a purely software approach (obviously, a much preferred approach to hardware solutions) [1]. The method exploits the fact that the Kikuchi bands formed by secondary electrons would have parallel edges if captured on a sphere rather than on the flat phosphor. If the correct pattern center were known, and the bands from the phosphor were mapped back onto a sphere, the bands would be parallel.

The method has been tested on various materials, including the EBSD pattern from a germanium sample, as shown in Fig. 1. In this example, two bands are chosen, and the approximate pattern center adjusted to obtain maximum intensity between planes that cut through parallel circles on the sphere, bounding these bands. The accuracy is better than $1/10^{th}$ of a pixel (as measured on the phosphor), and hence satisfies the requirements of HROIM. The method will also be extremely useful for regular OIM.

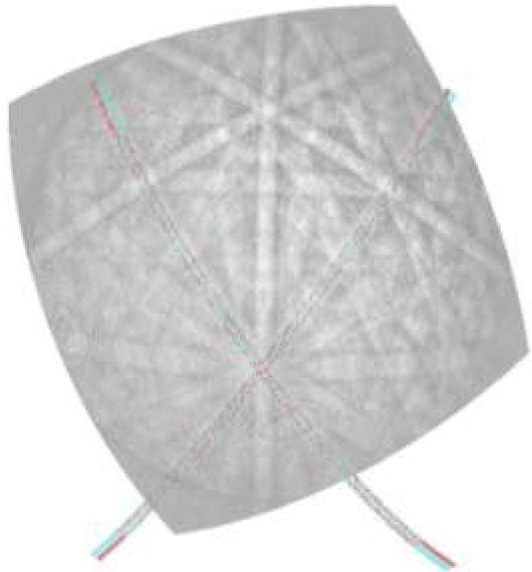


Figure 1. EBSD pattern from germanium sample mapped onto a sphere. The pattern center is adjusted to give the highest total intensity between parallel bands that overlay the original pattern.

2.2 The Simulated Pattern Method

Perhaps the most important advance achieved by the BYU team is the so-called "simulated pattern" augmentation of the Wilkinson cross-correlation method. In 2006 Wilkinson reported using cross correlations between adjacent electron backscatter diffraction (EBSD) images as a means to recover components of the elastic strain and rotation tensors. In this process the EBSD image is divided into multiple regions of interest (ROI). ROIs from two images are then compared by image cross correlation, to find the small shifts of features from one image to the other. In the Wilkinson method two experimental images are compared – the first from a 'strain-free' reference sample, and the second from the deformed sample. Cross correlations can be performed if the lattice misorientation between these two samples is ~ 1^{o} or less. The small displacements between the corresponding ROIs of these two images are recorded, and from these estimates of the elastic displacement gradient tensor can be recovered. Wilkinson's method is capable of resolving lattice orientation to within ~ 0.005^{o} and elastic strain to ~ 0.0001. But the method is severely limited by the need for a strain-free reference pattern, of nearly the same lattice orientation as the second pattern.

The BYU team considered simulated EBSD patterns as a reference, to take the place of the strain-free reference pattern. Kinematic Bragg's Law simulations were used to place the diffraction bands within the simulated image. Intensities were assigned to these bands, uniformly, according to the square of the structure-factor. The results were expressed by an angular resolution ~ 0.02^{o} and strain resolution of ~ 0.0004, when the pattern center is located with ~ 1/10 pixel precision. These results, while not quite as impressive as the Wilkinson recoveries, have much broader application because they are completely separated from the strain-free reference requirement.

These developments were described and published in a seminal article in Ultramicroscopy [2], and have been receiving considerable attention. Perhaps the most important feedback on the method is the agreement developed between BYU and TSL/EDAX to commercialize the method. The algorithms are currently protected via a full patent application in cooperation with TSL/EDAX.

2.2.1 HREBSD Resolution

The BYU team has worked with TSL to produce commercially-ready code, and has recently tested the performance of the code against standard OIM error metrics. Figure 2 demonstrates the clear superiority of the HREBSD code over regular OIM. Furthermore, when the newly developed pattern center algorithm is used to determine microscope geometry the improvement is dramatic. The orientation spread metric exhibits better than an order of magnitude error reduction [3, 4].

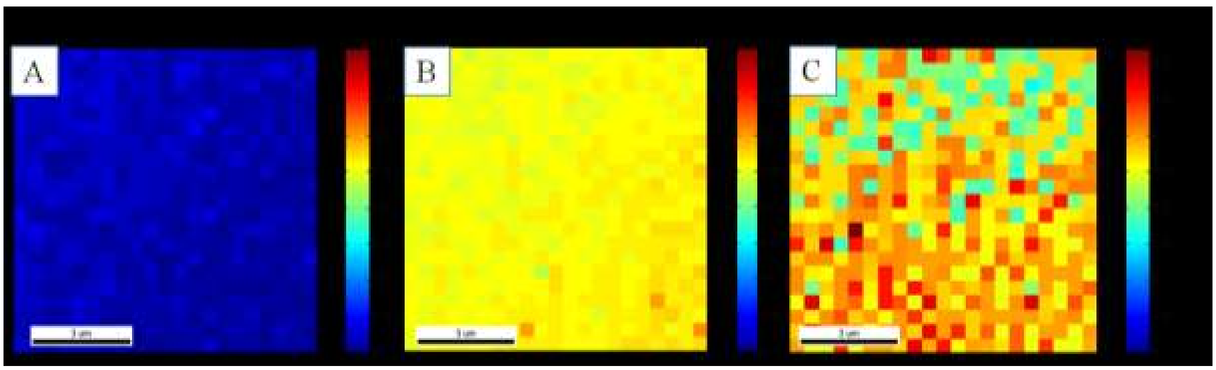


Figure 2. Misorientation maps (in degrees) from the average orientation of the PC-calibrated HR-EBSD scan A) HR-EBSD scan with PC calibration. B) HR-EBSD scan without PC calibration. C) Standard OIM scan.

2.2.2 Pattern center code validated and applied to strain measurements

Using the HREBSD approach with the PC determination algorithm, accurate strain measurements are now possible (Fig. 3). Such measurements are only possible via the HREBSD tools (with simulated bands) developed under this program [1, 3].

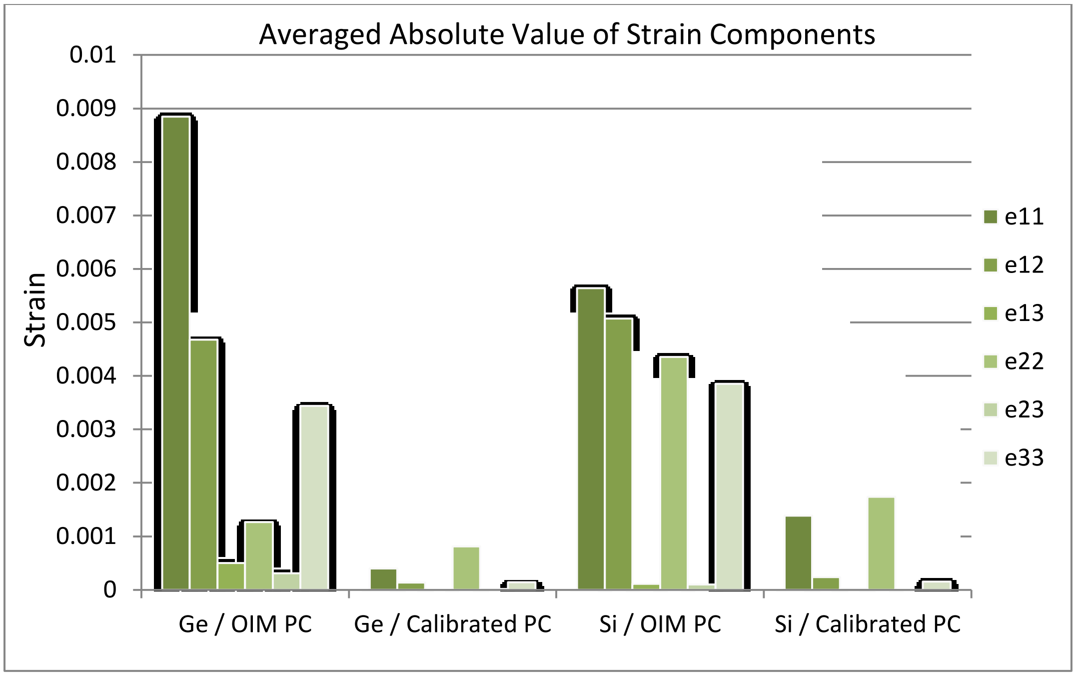
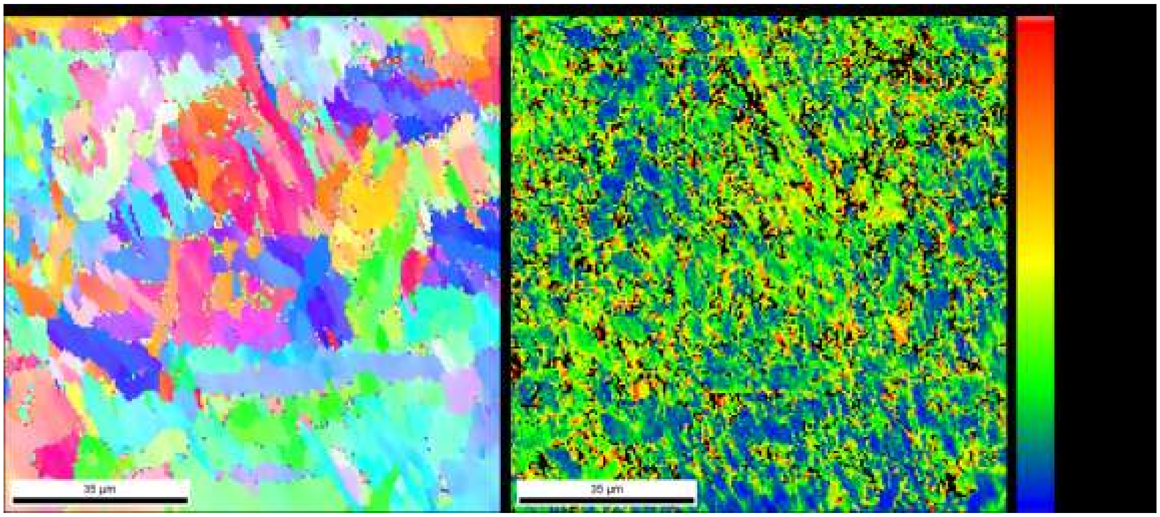


Figure 3. Average of the absolute value of all strain components for Si and Ge HR-EBSD scans using simulated reference patterns with and without a pattern center correction.

2.3 Recovery of Lattice Tetragonality and Pseudo-Symmetry Resolution

The simulated pattern method has been used to recover lattice tetragonality in high-strength low-alloy steels. Since the level of tetragonality can be small, it is difficult to distinguish between the tetragonal phases from the parent phase of higher symmetry. This is known as the "pseudo-symmetry" problem. Fe-C materials provide an ideal test-bed for the simulated pattern method. The challenge is to detect the effects of carbon distribution as it relates to the presence of Bainite phase (with small tetragonality) interspersed among the cubic ferrite. An example of the results is shown in Fig. 4, for two different HSLA steels processed by friction stirring. The range of tetragonality lies in the range 0 – 0.06. The higher average tetragonality is found in the L80 steel, and the largest tetragonalities are found near grain boundaries, where carbon will preferentially segregate. The view offered by these high resolution methods differs from what has been considered before: grains thought to be Bainite phase are actually, and typically, regions with varying tetragonality across a single grain.



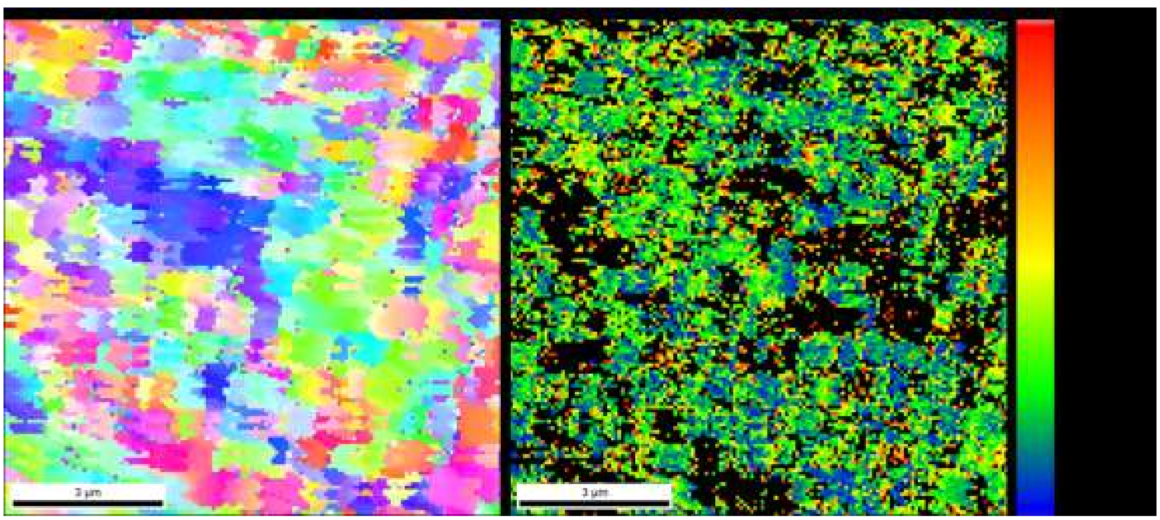


Figure 4. Inverse pole figure maps (left) and tetragonality map (right) from the HSLA 65 (top) and L80 (bottom) samples.

2.4 High accuracy dislocation density information extracted via HROIM

By using the full elastic distortion information available via HREBSD, a significantly improved estimate of dislocation density can be obtained (Fig. 5) [5, 6]. Using HREBSD together with in-situ scans of Mg, the first indications of twin formation at GBs, with correlation to dislocation activity, were observed, and noted by Beyerlein et al. in a recent paper concerning twin mechanics [7] (Fig. 6)

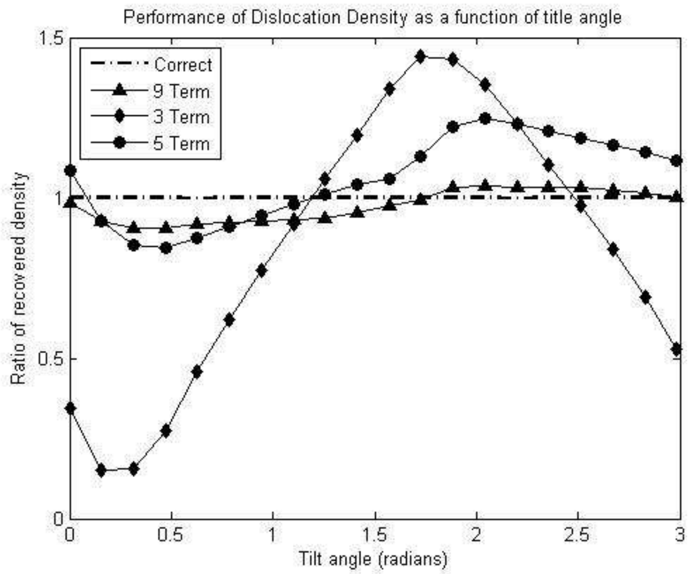


Figure 5. Recovery of simulated dislocation field via the standard 3-term or 5-term approaches, and the improved 9-term approach available from HREBSD

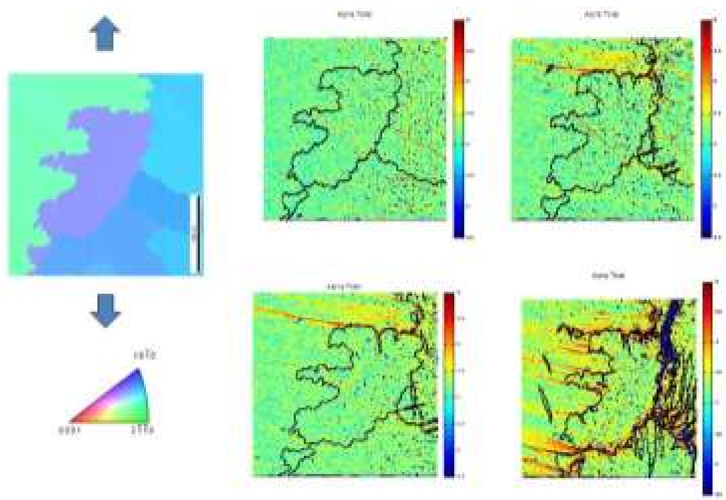


Figure 6. IPF orientation map of MgCe, and dislocation maps of in-situ tests at increasing strain levels. Twin nucleation is apparent, and appears to correlate with dislocation activity

2.5 Determination of full Nye tensor and relative activity

The stress equilibrium equations, implemented via a Green's function approach, have been utilized to extract the full dislocation density tensor from only the measurable 2D fields, on simulated examples (Fig. 7; some of this work was covered by an NSF EAGER award) [8].

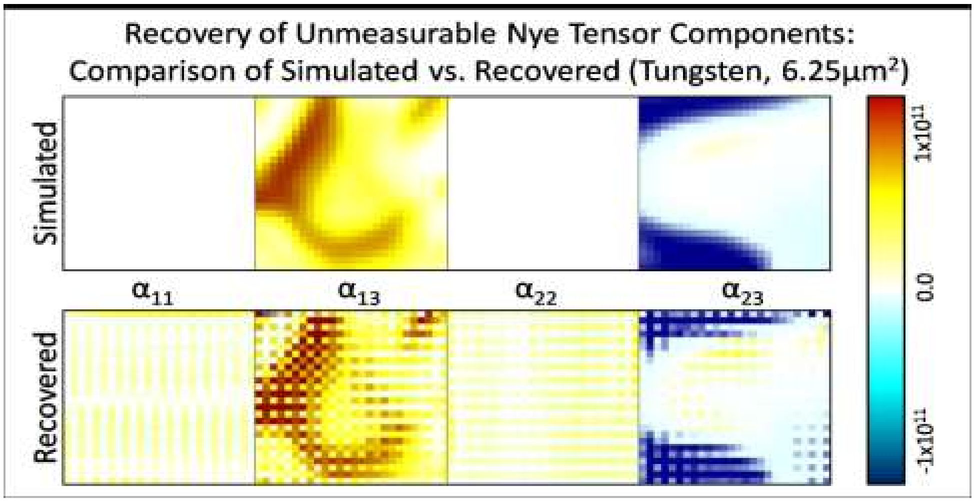


Figure 7. Demonstrating the capability of utilizing the stress equilibrium relations to recover the 4 non-measurable components of the Nye dislocation tensor, α.

A new technique for recovering relative activity on the various available slip systems from a knowledge of the Nye tensor has been implemented on simulated dislocation fields, resulting in superior extraction of the components. Figure 8. demonstrates extraction of relative activity in Mg using a more traditional method. The new technique is currently being coded for application to real data.

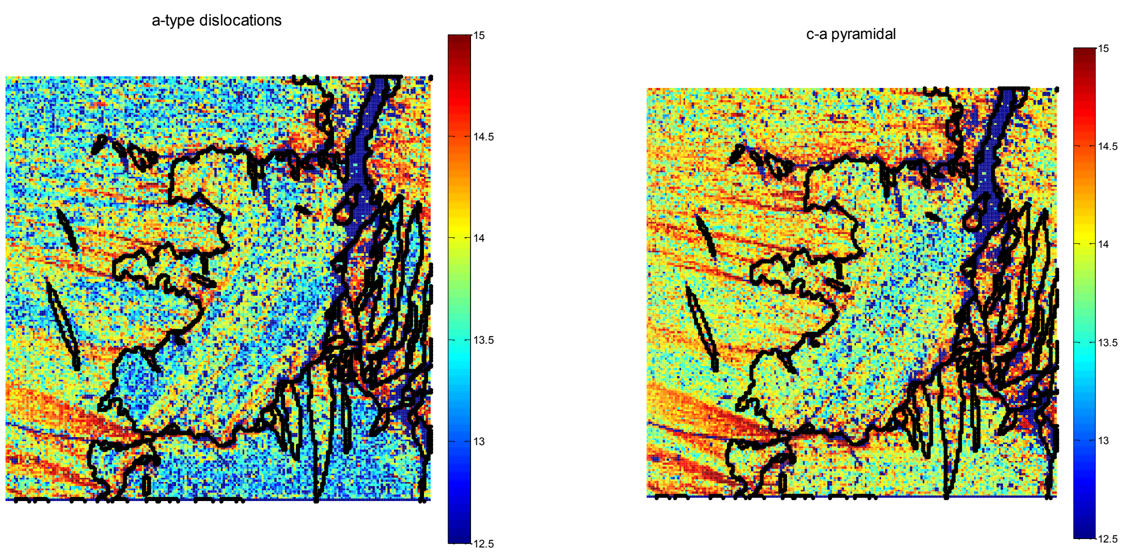


Figure 8. Relative activity of a-type and c+a-type dislocations in deformed MgCe

2.6 Spatial Resolution and Grain Boundary Analysis

The limits in spatial resolution of OIM are determined by the size of the interaction volume of the electron beam with the sample. When multiple lattice orientations lie within the interaction volume a convoluted EBSD pattern is produced, relating to all crystal lattice states within the volume. In order to improve spatial resolution of HROIM data, an accurate determination of the interaction volume is required (including quantification of the number of electrons arriving at the phosphor screen from each segment of the volume).

Monte Carlo modeling has been undertaken (using CASINO from the University of Sherbrooke), and an accurate map of the interaction volume obtained (Fig. 9). This has initially been used to deconvolve overlapping EBSD patterns at grain boundaries. Figure 10 shows the relative intensity of EBSD patterns from either side of a 60^{o} grain boundary as the electron beam passes the boundary, using this deconvolution method. Not only does this approach give us better spatial resolution from the original 2-D data, but we can potentially extract 3-D information regarding grain boundary inclination from the HROIM data.

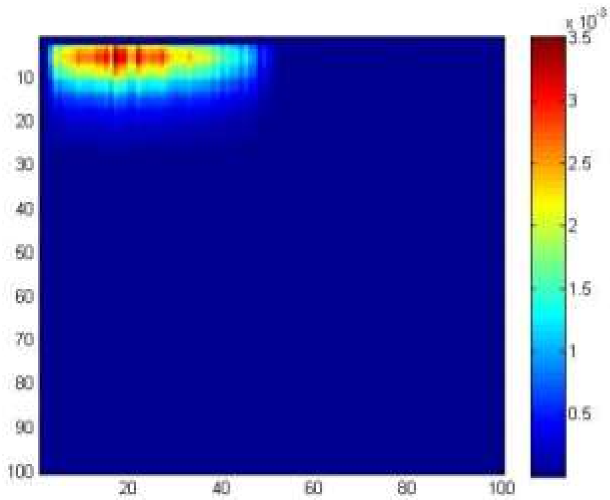


Figure 9. Cross section of interaction volume in Al (axes are in nm, and the scale is the normalized density of electrons from a given point)

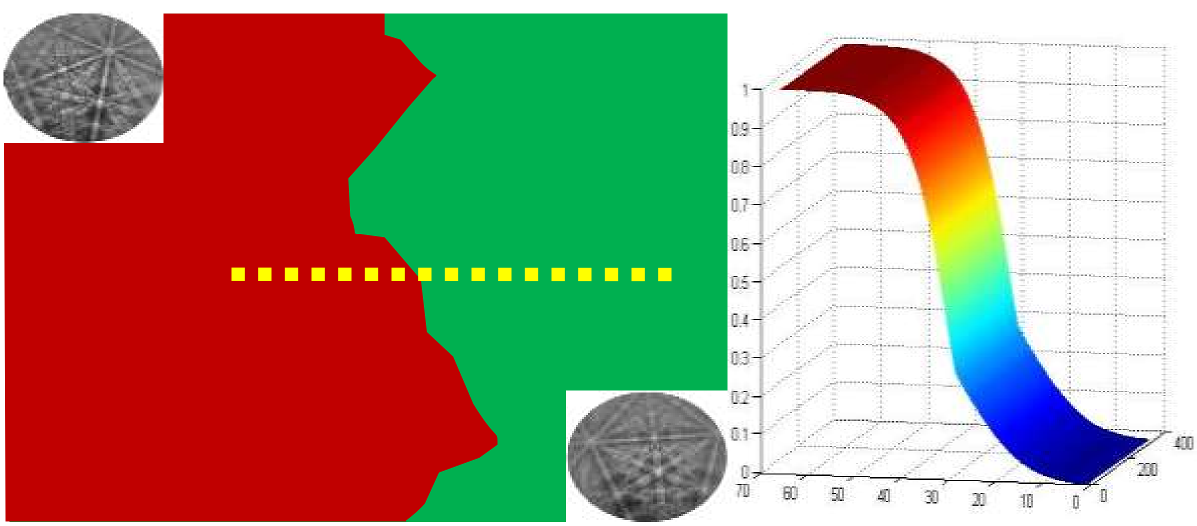


Figure 10. Left: Schematic of electron beam (yellow) crossing a 60^{o} grain boundary (the inclination is into the page, and hence is not shown); EBSD patterns from each grain are shown inset. Right: Relative intensity of each grain's pattern on the phosphor screen in a rectangle straddling the boundary (x-y scale in nm).

An internally developed Monte Carlo simulation approach to modeling the interaction volume within a sample has now been adopted, and libraries of curves are being developed to determine grain boundary inclination. Initial validation against 3D data, and twin boundaries (with known inclinations) indicate that the framework will resolve GB inclination to reasonable accuracy (Fig. 11) [9, 10].

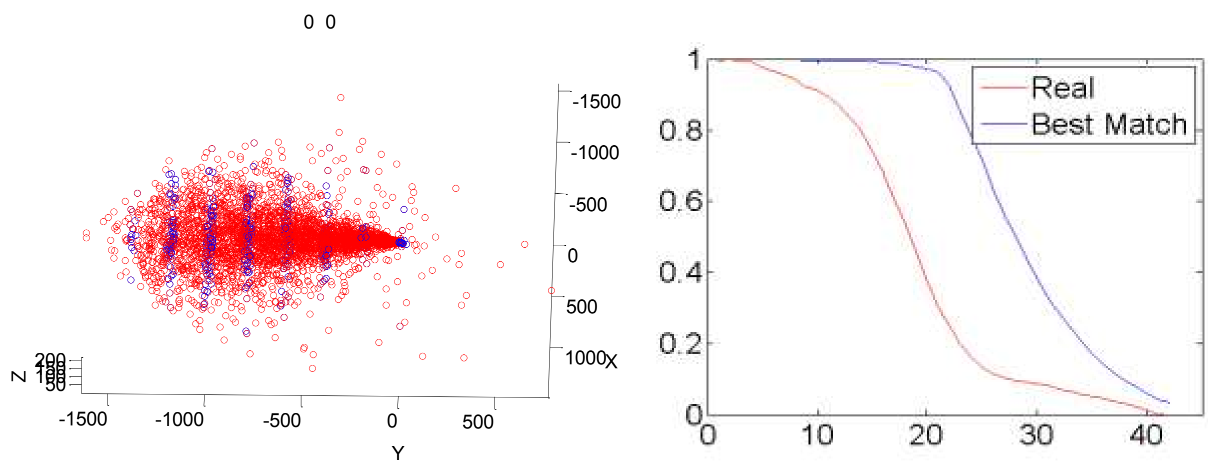


Figure 11. Model of electron interactions with a sample, and comparison of resultant pattern strength across a GB (red) with a simulated library curve (blue) in order to determine GB angle

2.7 Commercial-ready HROIM Code

Code has been developed in cooperation with TSL-EDAX that will provide the functionality of the HROIM framework to the scientific community. The screenshots in Fig. 12 illustrate the Matlab version of the code. The TSL version is even higher quality.

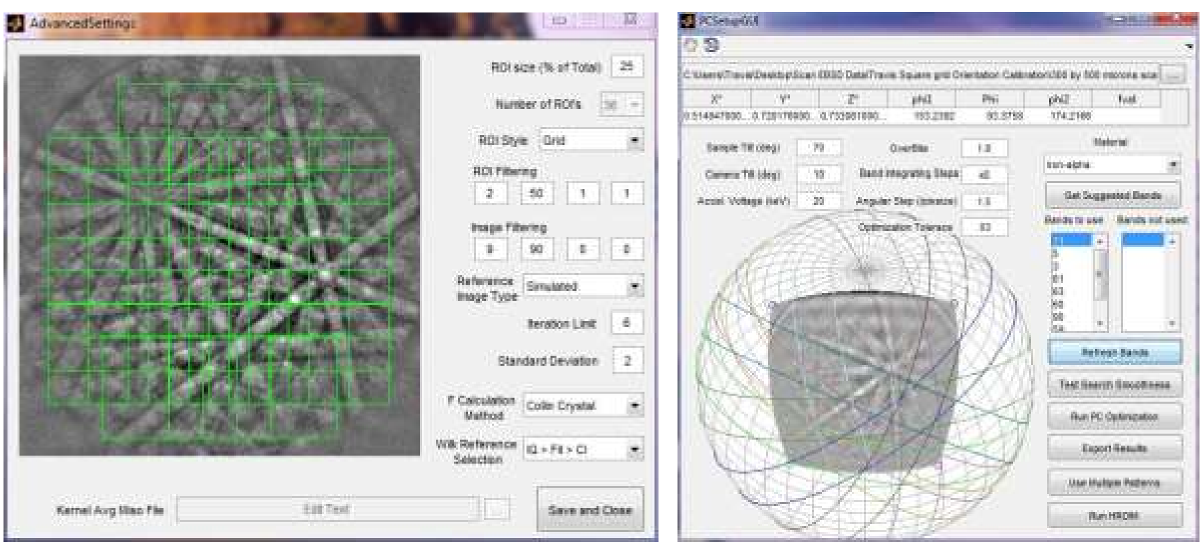


Figure 12. Screenshots from HROIM software (left) and pattern center calibration code (right)

2.8 Conductive Nano-Composites

In order to train several promising new undergraduates in the tools required for this project, parallel work was conducted on conductive nano-composites. Using focused ion beam and SEM microscopy the structure of the composite has been investigated and a mathematical model of properties developed, based upon a percolation-tunneling approach (Fig. 13). A novel method of determining nano-scale electrical properties (using a nano-indenter) has been proposed, and a wide range sensor application has been developed in partnership with LANL. Three journal papers have been published by this team [11-13], and eight conference presentations have been made by the undergraduates (including four refereed papers).

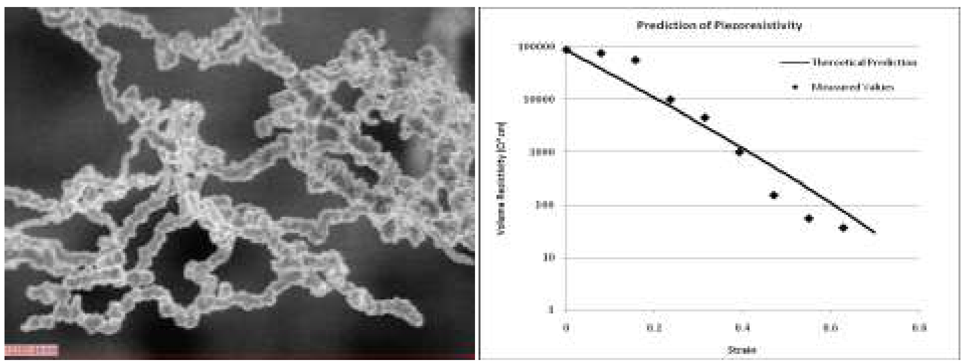


Figure 13. (Left) SEM image of nano-nickel used in conductive composite. (Right) Measured and predicted values of resistivity vs strain using percolation-tunneling model.

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